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Indian Standard
SPECIFICATION FOR ENDOSULFAN DUSTING POWDERS

(First Reprint JULY 1982)

UDC 632.95 Endosulfan

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR ENDOSULFAN DUSTING POWDERS

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(*Continued on page 2*)

INDIAN STANDARDS INSTITUTION
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NEW DELHI 110002

AMENDMENT NO. 4 JULY 1994
TO
IS 4322 : 1967 SPECIFICATION FOR ENDOSULFAN DUSTING POWDERS

(*Page* 5, *clause* 4.1) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause 2.2.1 of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR ENDOSULFAN DUSTING POWDERS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 October 1967, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Endosulfan dusting powders containing varying percentages of endosulfan, technical, are largely used in the control of pests of agricultural importance.

0.3 Taking into consideration the views of producers, consumers, testing authorities and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the manufacturing and trade practices followed in the country in this field.

0.4 This standard is one of a series of Indian Standards on pesticides and their formulations.

0.5 This standard contains clauses **3.1**, **E-2.3** and **E-3.4** which call for an agreement between the purchaser and the vendor.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or cal-

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a powder, shall be free flowing and devoid of any lumps; endosulfan used in its manufacture shall have been uniformly mixed in a suitable filler. The material, when dusted from a hand rotary duster, shall issue freely without clogging or bridging.

2.1.1 Endosulfan, technical, employed in the manufacture of this material shall conform to IS: 4344-1967*.

2.2 The material shall comply with the requirements as specified in Table 1.

TABLE 1 REQUIREMENTS FOR ENDOSULFAN DUSTING POWDERS

Sl No.	Characteristic	Requirement	Method of Test (Ref to Appendix)
(1)	(2)	(3)	(4)
i)	Endosulfan content, percent by weight	Nominal value as declared on the container (with tolerance of ±5 percent of the nominal value). (In case the nominal value declared is 5·0 percent or below, the tolerance shall be $^{+10}_{-5}$ percent of the nominal value.)	A
ii)	Sieving requirement, material passing through 75-micron IS Sieve*, percent by weight, *Min*	98	B
iii)	Bulk density after compacting	Not to exceed the value obtained before compacting by more than 60 percent.	C
iv)	Acidity (as H_2SO_4), percent by weight, *Max*	0·025	D
	OR		
	Alkalinity (as Na_2CO_3), percent by weight, *Max*	0·025	D

*See IS : 460-1962 'Specification for test sieves (*revised*)'. BS Sieve 200, ASTM Sieve 200 and Tyler Sieve 200 have their apertures within the limits specified for the above IS Test Sieve and may, therefore, be used in place of 75-micron IS Sieve.

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*Specification for endosulfan, technical.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel lined with a polyethylene liner; tinplate internally coated with corrosion resistant lacquer; polyethylene lined fibreboard or wooden containers; or hessian bags, polyethylene lined or bituminized, as agreed to between the purchaser and the vendor.

3.2 Marking — The containers shall be closed and sealed securely and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net weight of the contents;

f) Nominal content of endosulfan, technical, percent by weight; and

g) Minimum cautionary notice worded as under:

> 'AVOID CONTACT WITH SKIN AND INHALATION OF DUSTS. KEEP FAR APART FROM FOODSTUFFS AND FOODSTUFF CONTAINERS. DO NOT USE THIS CONTAINER FOR FOOD STORAGE.'

3.2.1 When hessian bags are used for packing this material, the pictorial marking for 'USE NO HOOKS. DO NOT PUNCTURE', as contained in IS : 1286-1967*, shall be stencilled on the bags.

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mrrk is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix E.

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*Pictorial markings for handling of goods in general (*first revision*).

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ENDOSULFAN CONTENT

A-1. PRINCIPLE OF THE METHOD

A-1.1 The principle in determination of endosulfan is based on hydrolysis of endosulfan with methanolic sodium hydroxide and estimating the sodium sulphite (Na_2SO_3) formed iodimetrically.

A-2. REAGENTS

A-2.1 Methanol

A-2.2 Sodium Hydroxide Pellets

A-2.3 Phenolphthalein Indicator Solution — one percent (*w/v*) in rectified spirit (conforming to IS : 323-1959†).

A-2.4 Standard Sulphuric Acid — 5 N.

A-2.5 Standard Iodine Solution — 0·1 N.

A-2.6 Standard Sodium Thiosulphate Solution — 0·1 N.

A-2.7 Starch Indicator solution — 0·2 percent, freshly prepared.

A-3. PROCEDURE

A-3.1 Weigh accurately a quantity of the material containing about 0·5 g of endosulfan, technical, and transfer it to a 250-ml flask. Add 100 ml of methanol by a pipette. Introduce about 1·5 g of sodium hydroxide pellets (about 15 pellets). Connect the flask to a reflux condenser and boil the contents at least for two hours. Shake the flask

---

*Specification for water, distilled quality (*revised*).

†Specification for rectified spirit (*revised*).

occasionally. Wash down the condenser with 20 ml of methanol and 50 ml of distilled water. Add one drop of the phenolphthalein indicator solution. Neutralize with standard sulphuric acid. Add a few drops of dilute sodium hydroxide solution to restore the indicator colour and to prevent any loss of sulphur dioxide.

A-3.2 For accurate determination of the sulphite, it is essential to add the sodium sulphite to an excess of acidified standard iodine solution and back titrate the excess of iodine with standard sodium thiosulphate solution.

A-3.3 Take 40 ml of standard iodine solution in a 500-ml glass stoppered iodine flask from a burette. Add 1 ml of the standard sulphuric acid and shake it slowly. While agitating, add the sulphite solution. Rinse the flask well with small portions of distilled water until all the sulphite has been transferred. (The washing procedure should be continued until there is insufficient sulphite in the flask to bleach a small drop of iodine.) The final volume in the iodine flask should be from 225 to 250 ml.

A-3.4 Back titrate the excess iodine with the standard sodium thiosulphate solution using starch solution as indicator and titrate until the blue colour disappears.

A-3.5 Carry out a blank determination by titrating 40 ml of the standard iodine solution with 175 ml of water acidified by 1 ml of standard sulphuric acid with standard sodium thiosulphate solution.

A-3.6 The difference in the volume of sodium thiosulphate solution indicates the iodine equivalent consumed by endosulfan.

A-4. CALCULATION

A-4.1 Endosulfan content, percent by weight $= \frac{V \times 2{\cdot}035}{W}$

where

V = volume in ml of 0·1 N iodine solution consumed by endosulfan (**A-3.6**), and
W = weight in g of the material taken for the test (**A-3.1**).

APPENDIX B

[*Table* 1, *Item* (ii)]

TEST FOR SIEVING REQUIREMENT

B-1. APPARATUS

B-1.1 Test Sieve — 75-micron IS Sieve, prepared for test by removing any film, grease or other water repellent material and then drying.

B-2. PROCEDURE

B-2.1 Weigh accurately 10·0 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a Ro-Tap or a similar machine for 20 minutes. Two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking up of any soft lumps of the caked material. After 20 minutes stop the machine and brush the residue on the sieve into a tared weighing dish Weigh the dish and determine the weight of the residue.

B-3. CALCULATION

B-3.1 Material passing through 75-micron IS Sieve, percent by weight $= 100 \left(1 - \frac{w}{W} \right)$

where

w = weight in g of the material retained on the test sieve, and

W = weight in g of the material taken for the test.

APPENDIX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF BULK DENSITY AFTER COMPACTING

C-1. APPARATUS

C-1.1 Graduated Cylinder — of 100-ml capacity with internal diameter of 27 to 29 mm.

C-1.2 Funnel — wide and short-stemmed.

C-2. PROCEDURE

C-2.1 Bulk Density Before Compacting — Rest the funnel over the top edge of the tared graduated cylinder. Fill the cylinder to the 100-ml mark by pouring the material through the funnel, without tapping, and level the powder with the minimum of disturbance. Leave the cylinder untouched for five minutes and add more powder, if necessary, to bring the contents to the 100-ml mark, levelling again with the minimum of disturbance. Weigh the filled cylinder and calculate the bulk density before compacting (weight of the material/volume occupied by the material).

C-2.2 Bulk Density After Compacting — Stopper the filled cylinder (C-2.1), and drop it 20 times through a height of 15 cm on to a felt pad resting on a hard surface. Note the volume of the material after compacting. Calculate the bulk density of the material after compacting (weight of the material/volume occupied by the material after compacting).

C-3. TEST EVALUATION

C-3.1 The value obtained in **C-2.2** shall not exceed the value obtained in **C-2.1** by more than 60 percent.

APPENDIX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF ACIDITY OR ALKALINITY

D-1. QUALITATIVE TEST

D-1.1 Procedure — Take about 0·5 g of the material in a test-tube, mix with about one millilitre of neutral acetone and warm gently to effect solution of the endosulfan present in the material. Cool, and add one millilitre of water to it and then test the solution for acidity or alkalinity with a litmus paper. Determine the acidity or the alkalinity, as the case may be, by the methods prescribed under **D-2** and **D-3**, respectively.

D-2. DETERMINATION OF ACIDITY

D-2.1 Reagents

D-2.1.1 *Acetone*

D-2.1.2 *Methyl Red Indicator Solution* — aqueous, one percent (*w*/*v*).

D-2.1.3 *Standard Sodium Hydroxide Solution* — 0·02 N.

D-2.1.4 *Standard Hydrochloric Acid* — 0·02 N.

D-2.2 Procedure

D-2.2.1 Weigh accurately about 10 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect solution of the endosulfan present in the material. Cool, add 75 ml of distilled water, filter and then titrate immediately with the standard sodium hydroxide solution using methyl red as the indicator.

D-2.2.2 Carry out a blank determination using 25 ml of acetone diluted with 75 ml of water.

D-2.3 Calculation

$$\text{Acidity (as } H_2SO_4\text{), percent by weight} = \frac{4{\cdot}9\,(V - v)\,N}{W}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

D-2.3.1 In case the blank shows alkaline reaction, neutralize with the standard hydrochloric acid and calculate as follows:

$$\text{Acidity (as } H_2SO_4\text{), percent by weight} = \frac{4{\cdot}9\,(VN_1 + vN_2)}{W}$$

where

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

N_1 = normality of the standard sodium hydroxide solution,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N_2 = normality of the standard hydrochloric acid, and

W = weight in g of the material taken for the test.

D-3. DETERMINATION OF ALKALINITY

D-3.1 Reagents

D-3.1.1 *Acetone*

D-3.1.2 *Methyl Red Indicator Solution* — aqueous, one percent (*w/v*).

D-3.1.3 *Standard Hydrochloric Acid* — 0·02 N.

D-3.1.4 *Standard Sodium Hydroxide Solution* — 0·02 N.

D-3.2 Procedure

D-3.2.1 Weigh accurately about 10 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect solution of the endosulfan present in the material. Cool, add 75 ml of distilled water, filter and then titrate immediately with the standard hydrochloric acid using methyl red as the indicator.

D-3.2.2 Carry out a blank determination using 25 ml of acetone diluted with 75 ml of water.

D-3.3 Calculation

$$\text{Alkalinity (as } Na_2CO_3\text{), percent by weight} = \frac{5{\cdot}3\,(V - v)\,N}{W}$$

where

V = volume in ml of the standard hydrochloric acid required for the test with the material,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N = normality of the standard hydrochloric acid, and

W = weight in g of the material taken for the test.

D-3.3.1 In case the blank shows acid reaction, neutralize with the standard sodium hydroxide solution and calculate as follows:

$$\text{Alkalinity (as } Na_2CO_3\text{), percent by weight} = \frac{5{\cdot}3\,(VN_1 + vN_2)}{W}$$

where

V = volume in ml of the standard hydrochloric acid required for the test with the material,

N_1 = normality of the standard hydrochloric acid,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N_2 = normality of the standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

APPENDIX E

(*Clause* 4.1)

SAMPLING OF ENDOSULFAN DUSTING POWDERS

E-1. GENERAL PRECAUTIONS

E-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

E-1.1 Samples shall not be taken in an exposed place.

E-1.2 The sampling instrument shall be clean and dry when used.

E-1.3 Proper precautions shall be taken while drawing samples since the material is toxic.

E-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the receptacles for samples from adventitious contamination.

E-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or by any other suitable means so as to bring all portions into uniform distribution.

E-1.6 The samples shall be placed in suitable, clean, dry and air-tight receptacles on which the material has no action.

E-1.7 The sample receptacles shall be of such a size that they are almost, but not completely, filled by the sample.

E-1.8 Each sample receptacle shall be sealed air-tight after filling and marked with full details of sampling, date of manufacture, name of the manufacturer and other particulars of the consignment.

E-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

E-1.10 Samples shall be protected from light.

E-2. SCALE OF SAMPLING

E-2.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

E-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

E-2.2 The number (*n*) of containers to be chosen from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

E-2.3 These containers shall be chosen at random from the lot and in order to ensure the randomness of selection, some random number table as agreed to between the purchaser and the vendor shall be used. In case such a table is not available, the following procedure shall be adopted:

> Starting from any container in the lot, count them as 1, 2, 3, etc, up to *r*, in one order, where *r* is equal to the integral part of the

value *N*/*n*, *N* being the total number of containers in the lot and *n* the number of containers to be chosen (Table 2). Every *r*th container thus counted shall be separated until the requisite number of containers is obtained from the lot to give samples for test.

TABLE 2 NUMBER OF CONTAINERS TO BE CHOSEN FOR SAMPLING

(*Clauses* E-2.2 *and* E-2.3)

LOT SIZE (*N*) (1)	NUMBER OF CONTAINERS TO BE CHOSEN (*n*) (2)
3 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
Over 110	10

E-3. TEST SAMPLES AND REFEREE SAMPLES

E-3.1 Before drawing the test sample, mix thoroughly the contents of each container selected, by shaking or by any other suitable means. Draw small portions of the material from different parts of each container selected (Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in Table 1 and shall be not less than 500 g.

E-3.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken for each selected container and shall be well mixed together so as to form a composite sample of not less than 60 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the vendor and the third for the referee.

E-3.3 The remaining portions of the material from each container (after a small quantity needed for the formation of the composite sample has been taken out) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried sample receptacles which are then sealed air-tight and labelled with all the particulars of sampling given under **E-1.8**. The material in each such sealed receptacle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each container selected (Table 2). One of these three sets shall be marked for the purchaser, another for the vendor and the third for the referee.

E-3.4 Referee Samples — Referee samples shall consist of the composite sample (**E-3.2**) and a set of individual test samples (**E-3.3**) marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the two.

E-4. NUMBER OF TESTS

E-4.1 Tests for description and the determination of endosulfan content, material passing through 75-micron IS Sieve, and bulk density after compacting shall be conducted individually on each of the samples constituting a set of test samples (**E-3.3**).

E-4.2 Test for the determination of the remaining characteristic, namely, acidity or alkalinity, shall be conducted on the composite sample as prepared under **E-3.2**.

E-5. CRITERIA FOR CONFORMITY

E-5.1 A lot shall be declared as conforming to the specification, when:

a) each of the test results for description, endosulfan content, material passing through 75-micron IS Sieve, and bulk density after compacting satisfies the corresponding requirement specified in Table 1. If, however, one or more of the test results do not satisfy the specified requirements, the conformity of the lot shall be ascertained in accordance with **E-5.1.1**; and

b) the test result on the composite sample for acidity or alkalinity satisfies the corresponding requirement given in Table 1.

E-5.1.1 In case one or more of the test results do not satisfy the requirements for endosulfan content, material passing through 75-micron IS Sieve and bulk density after compacting the following procedure shall be adopted for determining conformity of the material in respect of these characteristics. The mean and the range of the corresponding test results shall be calculated as:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range (R) = The difference between the highest and the lowest value obtained for the test results.

The appropriate expressions as shown in col 6 of Table 3 shall be calculated. If the values of these expressions satisfy the relevant condition as given in col 6 of Table 3, the lot shall be declared to have satisfied the requirements for endosulfan content, material passing through 75-micron IS Sieve and bulk density after compacting.

TABLE 3 CRITERIA FOR CONFORMITY

(*Clause* E-5.1.1)

SL No.	CHARACTERISTIC	TEST RESULTS 1, 2, 3,......n	AVERAGE	RANGE	CRITERION FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)	(6)
i)	Endosulfan content, percent by weight	—	$\bar{X}_1$	R_1	($\bar{X}_1 \pm 0{\cdot}6\ R_1$) shall lie within the nominal value ±5 percent of the nominal value.
ii)	Material passing through 75-micron IS Sieve, percent by weight	—	$\bar{X}_2$	R_2	($\bar{X}_2 - 0{\cdot}6\ R_2$) $\geqslant$ 98
iii)	Bulk density after compacting	—	$\bar{X}_3$	R_3	($\bar{X}_3 + 0{\cdot}6\ R_3$) $\leqslant$ bulk density before compacting +60 percent of the bulk density before compacting.

(*Continued from page* 2)

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